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Neutron Irradiation-Induced Dimensional Changes in MEMS Glass Substrates

Clark L. Allred[1,2,3], Jeffrey T. Borenstein[1], Linn W. Hobbs[2]
[1.] The Charles Stark Draper Laboratory, Inc., 555 Technology Square, Cambridge, MA 02139-3563
[2.] Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA 02139-4307
[3.] Air Force Institute of Technology, 2950 P Street, Wright-Patterson AFB, OH 45433-7765

ABSTRACT

A study is made of radiation-induced expansion/compaction in Pyrex and Hoya SD-2 glasses, which are used as substrates for MEMS devices. Glass samples were irradiated with a neutron fluence composed primarily of thermal neutrons, and a floatation technique was employed to measure the resulting density changes in the glass. Transport of Ions in Matter (TRIM) calculations were performed to relate fast (~1 MeV) neutron atomic displacement damage to that of boron thermal neutron capture events, and measured density changes in the glass samples were thus proportionally attributed to thermal and fast neutron fluences. The trend for strain with thermal neutron fluence (n/cm^2) was found to be a linear compaction of -2.8×10^{-20} for Pyrex and -1.0×10^{-21} for Hoya SD-2. For fast neutron fluence, the trend for strain was also linear: -6.1×10^{-21} for Pyrex and -7.9×10^{-22} for Hoya SD-2. The measured radiation-induced compaction of Pyrex is found to agree with that of previous studies. To our knowledge, this work represents the first study of compaction in Hoya SD-2 with neutron fluence. Hoya SD-2 is of considerable importance to MEMS, owing to its close thermal expansivity match to silicon from 25-500 °C.

INTRODUCTION

In order for a glass substrate-mounted MEMS device to behave predictably when used in a radiation environment, one must design it with an understanding of the effect of that environment on material properties such as density, coefficient of thermal expansion, and elasticity. This study treats density changes caused by neutron irradiation which induce differential strains between a MEMS device and its substrate.

One mechanism for causing dimensional changes in glass is the collision of a fast neutron with atoms in the glass network structure. The atoms so struck can be forced from their network sites with substantial kinetic energy, in turn leading to other atomic displacements and subsequent network rearrangements. This sequence of atomic displacements is referred to as a collision cascade, and the atom directly displaced by an incident fast neutron is called the primary knock-on atom (PKA).

Collision cascades can also be caused by nuclear reactions that yield energetic particles. The reaction of interest here is the absorption of a thermal neutron by ^{10}B, an isotope 20% naturally abundant in boron and present in both Pyrex and Hoya SD-2. Upon absorbing a thermal neutron, the ^{10}B nucleus splits into a lithium nucleus with a kinetic energy of ~0.9 MeV, and an alpha particle with kinetic energy of ~1.6 MeV.[1] Both of these products can then initiate a collision cascade.

The effects of thermal neutron irradiation in Pyrex were studied by Paymal.[1] He employed a floatation technique called "sink/float" to measure small irradiation-induced density differences in several glass types. He observed a compaction rate with dose that at lower thermal neutron fluences ($< 1 \times 10^{17}$ n/cm^2) was approximately linear, with a slope of -2×10^{-20} strain per n/cm^2.

No studies of which we are aware involving fast neutron-induced strain have been made for either glass. Primak,[2] however, did report the initial compaction rate of vitreous silica with fast neutron exposure to be -5.6×10^{-22} strain per n/cm^2. Shelby's[3] results for gamma irradiation of vitreous silica show that SiO_2 compacts an order of magnitude less quickly with dose than does Pyrex. Compaction of Pyrex with gamma irradiation has been measured by Shelby,[4] Conners,[5] Sato,[6] and Zdaniewski.[7] Of these studies, Shelby's covers the greatest dose range. Shelby's data reveal an initially linear trend of compaction with dose of -1.4×10^{-13} strain per rad at doses up to $\sim2\times10^9$ rad (2×10^7 Gy). The effect begins to saturate at higher doses.

EXPERIMENT

Two glasses chosen principally for their close thermal expansion coefficient match with silicon, Pyrex® [8] and Hoya SD-2®,[9] were studied. The approach taken here included neutron irradiation of both glasses followed by density measurements using a floatation technique ("sink/float"). Since the neutron flux used contained both fast and thermal neutrons, Monte-Carlo simulations of thermal and fast neutron collision cascades were used to apportion induced strain between the two damage mechanisms.

Pyrex and Hoya SD-2 wafers approximately 0.78 mm thick were diced into 3 mm squares using a diamond saw. Sink/float compares the density of one material sample to that of another. Accordingly, each irradiated sample had an unirradiated control sample. To minimize the probability of the glass pieces within a sample pair having an initial density difference, care was taken to choose glass squares adjacent to each other on the same wafer. In anticipation of the need for distinguishing between the irradiated samples and their control samples, the control samples had a corner cut off. While some discoloration due to irradiation was expected, whether or not this would suffice for distinguishing the two samples during a sink/float measurement was not known.

Samples were irradiated by MIT nuclear reactor personnel in a pneumatic tube facility. Estimates furnished by reactor personnel of the neutron fluxes for samples within the facility are 1×10^{11} n/cm^2/s fast (1 MeV equivalent) and 6.7×10^{12} n/cm^2/s thermal. Samples were held within plastic tubes ("rabbits") that are 2.5 cm in diameter and about 6 cm long. The temperature during irradiation was not monitored, but did not exceed the softening point (~ 90 °C) of the polystyrene supports that held the samples within the rabbits, as evidenced by the lack of deformation in any of the supports upon withdrawal of the rabbits from the facility.

The ideas behind the sink/float technique are primarily drawn from Knight[10], Shelby,[3] and Conners.[5] The method has also been used by Sato[6] and Zdaniewsky,[7] Paymal,[1] and again by Shelby.[4] Shelby and Conners report the greatest precision for the technique, with Shelby[4] reporting 50 part per million (ppm) precision for $\Delta\rho/\rho$, Conners reporting ~90 ppm precision, and Sato claiming 300 ppm precision.

To perform a sink/float measurement, a pair of glass samples (one irradiated, one not) is placed in a test tube containing a heavy liquid in which they just float. The liquid is then heated by means of a water bath. If the starting density of the liquid is sufficiently close to that of the

glass, a few degrees of heating will cause the liquid density to become smaller than that of the glass. The glass samples sink at different temperatures if their densities are different. By knowing the thermal expansion coefficient for the liquid, one can relate the difference in sinking temperatures to a density difference.

An approximate formula that relates a difference in sinking temperature to a density difference is:

$$\frac{\Delta\rho}{\rho} = \frac{3\Delta T(\alpha_L - \alpha_G)}{1 - 3\Delta T\alpha_G} \qquad (1)$$

In Equation 5.1, ΔT is the difference in sinking temperature between the irradiated and unirradiated glass samples, α_G is the linear coefficient of thermal expansion of the glass, α_L is the linear coefficient of thermal expansion of the liquid (a third of the volumetric coefficient of thermal expansion), and ρ is the glass density. Since the thermal expansion of the glass is $\sim 10^{-6}$ K^{-1} and that of the liquid is $\sim 10^{-4}$ K^{-1}, a good approximation can be made simply by:

$$\frac{\Delta\rho}{\rho} = 3\Delta T\alpha_L \qquad (2)$$

Assuming an uncertainty of 0.01 °C in each temperature reading and neglecting the uncertainty in the coefficient of thermal expansion for the liquid, one obtains a predicted uncertainty in a strain calculation ($\varepsilon = -(1/3)\Delta\rho/\rho$) of about 6 ppm.

Six heavy liquid-filled 38 mm o.d. × 200 mm Pyrex test tubes were suspended from holes in a plexiglass lid that were cut in a regular circular pattern. The lid rested on a cylindrical 25 cm o.d. × 25 cm Pyrex jar. An immersion heater was suspended from a hole in the center of the lid and attached to a transformer. The jar was filled with water to above the fluid level in the test tubes, and a stir bar was used at the bottom of the jar. A horizontal line was drawn about 5 cm up from the bottom of each test tube. A platinum resistance temperature detector probe was inserted through a rubber stopper and immersed in the liquid of one of the test tubes. The probe was attached to a precision thermometer with a 0.01 K readout. The heavy liquids used were tetrabromoethane (also known as acetylene tetrabromide) and bromonaphthalene. The first was suspected of being quite hazardous, so all work was performed in a vent hood while wearing goggles and a respirator with organic vapor filter cartridges. The two liquids were mixed to produce densities slightly superior to the nominal 2.23 g/cm^3 and 2.60 g/cm^3 of the Pyrex and Hoya SD-2 samples, respectively.

The heavy liquid mixture was heated at a rate of 0.1 K per minute. A sample was declared as "sunk" when it crossed the line drawn on the test tubes. Six measurements were made for each glass sample pair. The averaged results are shown in Figure 1. The error bars shown represent the standard deviation of the six measurements divided by the square root of six, then averaged across all five sample pairs. This is the uncertainty for the mean value of the six measurements.[11] The value of α_L in equation 2 is taken from measurements by Conners.[5] Conners used sink/float to detect gamma-induced density changes in glasses with densities from 2.13 g/cm^3 to 2.52 g/cm^3. Her liquid mixtures were all composed of the same two chemicals used here, tetrabromoethane and bromonaphthalene. She observed that the volumetric thermal expansion for all of her liquids averaged to 840 ppm/K, with a standard deviation of 8 ppm/K.

Both thermal and fast neutron collision cascades involve similar PKA energies, and the majority of displaced atoms will be displaced by ions created from secondary collisions.

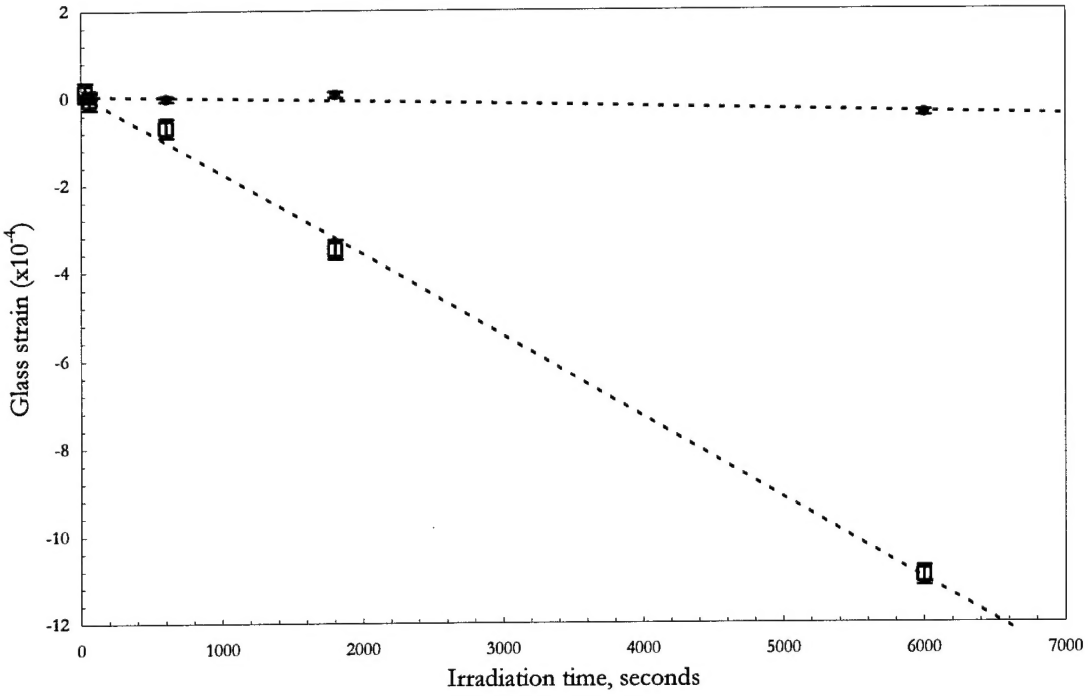

Figure 1. Strain in neutron-irradiated glass samples (relative to unirradiated samples). Neutron fluence was a mixture of fast and thermal neutrons.

Therefore, it seems reasonable to assume that the network rearrangement processes involved in both types of collision cascades will be similar, and that the dimensional change in a glass can be related directly to the number of atoms displaced in these collision cascades. To relate fast neutron-induced compaction to thermal neutron-induced compaction, we need an idea of how many displacements occur in each type of cascade. Monte-Carlo simulations using TRIM (version 2000.38)[12] were carried out for both thermal and fast neutron collision cascades in both Pyrex and Hoya SD-2. Inputs to TRIM included glass composition (obtained from XPS measurements) and the energy distribution of PKAs. Table 1 summarizes the results for a thin (~ 1mm) glass wafer.

Using the fast (1 MeV equivalent) and thermal fluences for the irradiation facility, linear fits to the results in Figure 1, and the results in Table 1, it is possible to estimate how much strain is expected in the glass samples for a given fluence of fast or thermal neutrons. These estimates are shown in Table 2.

Table 1. Prediction of atomic displacement events per incident neutron, calculated using TRIM.

	PYREX	HOYA SD-2
DISPLACEMENTS PER INCIDENT FAST NEUTRON (1 MEV)	22	24
DISPLACEMENTS PER INCIDENT THERMAL NEUTRON	100	31

Table 2. Strain as a linear function of thermal neutron fluence, fast neutron fluence, and displacements per atom (DPA).

	STRAIN/n/cm^2 THERMAL	STRAIN/n/cm^2 FAST (1 MEV)	STRAIN/ DPA
PYREX	-2.77×10^{-20}	-6.07×10^{-21}	-1.49
HOYA SD-2	-1.01×10^{-21}	-7.89×10^{-22}	-0.19

DISCUSSION

The error bars on data points in Figure 1 represent ±22 ppm for the Pyrex samples and ±6 ppm for the Hoya SD-2 samples. We attribute this difference in measurement scatter to the different liquid-sample interactions in the two mixtures used for the two glass types. It was observed during the measurements that the floating samples in both cases had an attraction to the liquid surface, and would therefore not always immediately sink when the liquid density became less than the glass sample density. This surface attraction was more noticeable between the Pyrex samples and the liquid mixture used for these than for the Hoya SD-2 samples.

The Hoya SD-2 was observed to undergo almost an order of magnitude less compaction than the Pyrex at a given fast neutron fluence. The response of Hoya SD-2 to thermal neutrons is even smaller in proportion to that of the Pyrex; however, this can be partly attributed to the smaller boron concentration in Hoya SD-2.

The values for fast neutron-induced compaction in Pyrex and Hoya SD-2 are found using an assumption of equivalent damage mechanisms for thermal and fast neutron collision cascades. We can test this assumption by comparing our results with those of other experiments and check for consistency. Our prediction for thermal neutron-induced strain in Pyrex is in good agreement with Paymal's[1] result. There are no data available for direct comparison with the fast neutron results; however, Shelby[3,4] demonstrated that Pyrex compacts by about an order of magnitude more than does vitreous silica for a given gamma dose. Since our prediction for Pyrex compaction with fast neutron fluence is also an order of magnitude larger than the result for vitreous silica,[2] we feel that this provides support (albeit indirect) for our assumption that the compaction caused by thermal and fast neutrons can be related by using the ratio of the number of displacements in both collision cascades.

CONCLUSIONS

The density change induced in Pyrex and Hoya SD-2 glasses by irradiation with neutrons has been measured using a sink/float technique. Monte-Carlo calculations of total displacements in neutron collision cascades were used to compare the compaction caused by fast neutrons with that caused by thermal neutron capture by boron present in the glasses. The magnitude of compaction observed in Pyrex that is attributable to thermal neutrons agrees well with a previous measurement. The radiation-induced compaction of Hoya SD-2 glass, here measured for the first time, is observed to be significantly lower than that of Pyrex, making it a good candidate as a substrate for MEMS applications involving radiation exposure.

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